

# Development of a Bulk Calcium Phosphate Ceramic Capable of Supporting Osteoclastic Resorption

In the past twenty years, there has been an increased interest in calcium phosphates and their role in surgical and dental implants. The search has been for materials that have no adverse physiological response, and will enhance the biocompatibility and long-term reliability of an implant. The ideal material would be totally resorbed by the body as it was replaced by natural bone. This material would be free of any substances of a biological origin which might elicit an immune response.

It is now commonly accepted that calcium phosphate based ceramics are capable of sustaining bone cell growth (osteogenic). Much attention has therefore been paid to these materials because of their potential in applications such as hip replacements and dental implants. Since calcium hydroxylapatite (HA),  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , is the calcium phosphate which most resembles the primary inorganic component of bone, metal implants used in joint replacements have been plasma sprayed with HA to increase the ingrowth of natural bone thus better cementing the implant. Investigators have been able to produce coatings of high quality, stoichiometric materials (such as HA). However these types of coatings have low resorption levels and if the plasma sprayed coating detaches, the resultant powder can cause inflammation in adjacent tissues.

A significant contribution for the advancement of these type of coatings was developed by Q.Qiu at Queen's University. Calcium phosphate based thin films prepared by colloidal sol-gel methods on quartz substrates were produced which are bioactive to both resorption and deposition of natural bone. Since these films are very thin (approximately 0.5 microns) and are prepared on transparent substrates, the holes, or resorption pits, created in the film by osteoclasts are visible under a light microscope. These films are not only useful for the advancement of implant coatings, but also as a direct means for quantifying cell resorption activity and for determining the mechanisms whereby cells interact with surfaces. This technology was transferred to Millenium Biologix Inc. and these films are now commercially available under the trade name Osteologic™.

Thin films are formed on quartz plates by coating from a particulate sol gel suspension. The basic reaction is:



Air dried films are sintered at 1000°C for one hour at ambient humidity. These bioactive films have a unique, porous multilayer, structure and composition on quartz: approximately 67% silicon stabilized alpha-tricalcium phosphate (alpha-TCP) and 33% HA. The macroscopic size of the granules which compose the coating is of the same order as that deposited by osteoblasts during new bone formation. Sintered films have a thickness of the order of less than 0.5 microns and a grain size of approximately 0.5 microns.

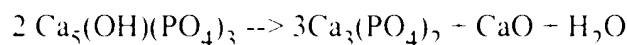


Figure 1: SEM Image of a Sintered Osteologic Film

Initial experiments, performed at Queen's using a new thermal processing unit designed to control the sintering temperature profile and environment, have determined that the phase transition from HA to

alpha-TCP in the film occurs very quickly and is dependent primarily on the sintering temperature. Since this transformation is not observed in the bulk material, the mechanism must be attributable to the dynamics of the thin film and interaction with the quartz substrate. These results are summarized in Langstaff, et al. *Mat. Res. Soc. Symp. Proc.* Vol **414** p 87-92.

The proposed dehydration mechanism is facilitated by the large surface area to volume ratio present in the porous films and interaction with the quartz substrate.



Suppression of the transformation in a humid environment has been observed and thermodynamic calculations, which confirm this hypothesis, have been performed using the Facility for the Analysis of Chemical Thermodynamics (FACT) database.

In S. Langstaff's recently completed PhD thesis, the thin film technology has been extended to include a bulk ceramic form. This bulk ceramic, Ostite<sup>TM</sup>, possesses the same phase composition and surface morphology as the thin film materials and has been shown to be bioactive to both osteoclast resorption and osteoblast deposition.

Further experimental work has indicated that the phase once thought to be alpha-TCP, is in fact a silicon stabilized tricalcium phosphate (Si-TCP) similar in crystallographic structure to alpha-TCP. Substitution of silicon at the phosphorous tetrahedral sites has been demonstrated through FTIR and XANES experiments. A paper describing the complete materials analysis of the Si-TCP has been submitted to the journal "Biomaterials".

Current projects involving this new bone-biomaterial include the extension of the bulk ceramic technology to create porous ceramics to be used as synthetic bone graft materials. This involves increasing the compression strength and inherent toughness of the porous material. Extension of the technology to include coatings on industrially relevant implant surfaces and the creation of bulk macroporous ceramics is currently underway. The effect of the films' structure and composition on bone cell growth is also being examined.

This project is being carried out under the supervision of Dr. Michael Sayer, Queen's University, and in collaboration with Dr. R. Smith in Materials and Metallurgical Engineering, Queen's University, and Millenium Biologix Inc. located in Kingston, Ontario.

## RECENT PUBLICATIONS

Langstaff S., Sayer M., Weaver L., Pugh S. & Smith T. *Mat. Res. Soc. Symp. Proc.* **414**, (1996) pp. 87-92.

Griswold E. & Langstaff S. **Transmission Electron Microscopy of Calcium Phosphate Thin Films.** (unpublished)

Davies C., Langstaff S., Sayer M., Smith T. & Pugh S. **Titanium Doping of Calcium Phosphates: Effect of Carrier and Dopant Concentration on Phase Composition and Ceramic Morphology.** (unpublished)

Langstaff S., Sayer M., Pugh S. & Smith T. **Resorbable Bioceramics based on Stabilized Calcium Phosphates - Part I : Rational Design, Preparation and Material Characterisation.** (submitted to Biomaterials)

Langstaff S., Sayer M., Pugh S., & Smith T. **Resorbable Bioceramics based on Stabilized Calcium Phosphates - Part II: Evaluation of Biological Response.** (submitted to Biomaterials)

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*Created by Sarah Langstaff (4sl@qlink.queensu.ca)*